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IS 1448-112 (1983): Methods of test for petroleum and its products, Part 112: Determination of lead in gasolsine by atomic absorption spectrometry [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 112]

DETERMINATION OF LEAD IN GASOLINE BY ATOMIC ABSORPTION SPECTROMETRY

UDC 662·753·12 : 543·422 : 546·815

Adapted with Permission from ASTM D 3237-79.

1. SCOPE

1.1 This method covers the determination of the total lead content of gasoline within the concentration range of 2·5 to 25 mg of lead/litre. The method compensates for variations in gasoline composition and is independent of lead alkyl type.

2. OUTLINE OF THE METHOD

2.1 The gasoline sample is diluted with methyl isobutyl ketone and the alkyl lead compounds are stabilized by reaction with iodine and a quarternary ammonium salt. The lead content of the sample is determined by atomic absorption flame spectrometry at 2 833 Å, using standards prepared from reagent grade lead chloride. By the use of this treatment, all alkyl lead compounds give identical response.

3. SIGNIFICANCE AND USE

3.1 This method is used to ensure compliance of trace lead for lead free gasoline.

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4. APPARATUS

4.1 Atomic Absorption Spectrometer — Capable of scale expansion and nebulizer adjustment, and equipped with a slot burner and premix chamber for use with an air-acetylene flame.

4.2 Volumetric Flasks — 50-ml, 100-ml, 250-ml and 1-litre sizes.

4.3 Pipettes — 2-ml, 5-ml, 10-ml, 20-ml and 50-ml sizes.

4.4 Micropipette — 100-μl, Eppendor type or equivalent.

5. REAGENTS

5.1 Aliquat 336* — Tricapryl methyl ammonium chloride.

5.2 Aliquat 336/MIBK Solution (10 percent v/v) — Dissolve and dilute 100 ml (88·0 g) of Aliquat 336 with MIBK to 1 litre.

5.3 Aliquat 336/MIBK Solution (1 percent v/v) — Dissolve and dilute 10 ml (8·8 g) of Aliquat 336 with MIBK to 1 litre.

5.4 Iodine Solution — Dissolve and dilute 3·0 g of iodine crystals with toluene to 100 ml [*Warning*: Flammable. Vapour harmful (*see* Appendix A)].

5.5 Lead Chloride

5.6 Lead-Sterile Gasoline† — Gasoline containing less than 1·25 mg Pb/l†. [*Warning*: Extremely flammable. Harmful if inhaled. Vapours may cause flash fire (*see* Appendix A)].

NOTE — To confirm lead concentrations of less than 1·25 mg Pb/l refer to Methods ASTM D 1363 and ASTM D 3116. A procedure for the purification of gas turbine fuel appears in Appendix A4 of Method ASTM D 2550 and may be useful in decreasing the lead concentration of low lead gasoline to lead-sterile gasoline.

5.7 Lead Standard Solution (1·25 g Pb/l) — Dissolve 0·4195 g of lead chloride ($PbCl_2$) previously dried at 105°C for 3 hours in about 200 ml of 10 percent Aliquat 336/MIBK solution in a 250-ml volumetric flask. Dilute to the mark with the 10 percent Aliquat solution, mix and store in a brown bottle having a polyethylene-lined cap. This solution contains 1·25 g Pb/l.

5.8 Lead, Standard Solution (250 mg Pb/l) — By means of a pipette accurately transfer 50·0 ml of the 1·25 g Pb/l solution to a 250-ml volumetric flask and dilute to volume with 1 percent Aliquat/ MIBK solution. Store in a brown bottle having a polyethylene-lined cap.

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*Available from General Mills Corporation, Minneapolis Minn. USA.

†Available from Amoco Oil Company, 200 East Randolph Street, Chicago, Ill 60601.

5.9 Lead, Standard Solution (5, 12·5 and 25 mg Pb/l) — Transfer accurately by means of pipettes 2·0, 5·0 and 10·0 ml of the 250 mg Pb/l solution to 100-ml volumetric flasks; add 5·0 ml of 1 percent Aliquat 336 solution to each flask; dilute to the mark with MIBK. Mix well and store in bottles having polyethylene-lined caps.

5.10 Methyl Isobutyl Ketone (MIBK), (4-methyl-2-pentanone)

6. SAMPLING

6.1 Collect sample in a metal container which can be sealed against leakage.

7. CALIBRATION

7.1 Preparation of Working Standards — Prepare three working standards and a blank using the 5, 12·5 and 25 mg Pb/l standard lead solution described in **5.9**.

7.1.1 To each of four 50-ml volumetric flasks containing 30 ml of MIBK, add 5·0 ml of low lead standard solution and 5·0 ml of lead-free gasoline. In the case of the blank, add only 5·0 ml of lead-free gasoline.

7.1.2 Add immediately 0·1 ml of iodine/toluene solution by means of the 100-μl Eppendorf pipette. Mix well.

7.1.3 Add 5 ml of 1 percent Aliquat 336 solution and mix.

7.1.4 Dilute to volume with MIBK and mix well.

7.2 Preparation of Instrument — Optimize the atomic absorption equipment for lead at 2 833 Å. Using the reagent blank, adjust the gas mixture and the sample aspiration rate to obtain an oxidizing flame.

7.2.1 Aspirate the 25 mg Pb/l working standard and adjust the burner position to give maximum response. Some instruments require the use of scale expansion to produce a reading of 0·150 to 0·170 for this standard.

7.2.2 Aspirate the reagent blank to zero the instrument and check the absorbances of the three working standards for linearity.

8. PROCEDURE

8.1 To a 50-ml volumetric flask containing 30 ml MIBK, add 5·0 ml of gasoline sample and mix.

8.1.1 Add 0·10 ml (100 μl) of iodine/toluene solution and allow the mixture to react for about 1 minute.

8.1.2 Add 5·0 ml of 1 percent Aliquat 336/MIBK solution and mix.

8.1.3 Dilute to volume with MIBK and mix.

8.2 Aspirate the samples and working standards and record the absorbance values with frequent checks of the zero.

9. CALCULATION

9.1 Plot the absorbance values *versus* concentration represented by the working standards and read the concentrations of the samples from the graph.

10. PRECISION

10.1 Results of duplicate tests shall not differ by more than the following amounts:

Repeatability	*Reproducibility*
1·25 mg Pb/l	2·5 mg Pb/l

APPENDIX A

(*Clause* 5.4 *and* 5.6)

PERCAUTIONARY STATEMENT

A-1. TOLUENE

Warning: Flammable, vapour harmful.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing of vapour of spray mist.
Avoid prolonged or repeated contact with skin.
In case of spillage soak up with sand or diatomaceous earth.
In case of fire use water spray, foam, dry chemical or CO_2.

A-2. GASOLINE (WHITE)

Danger: Extremely flammable.
Harmful if inhaled. Vapours may cause flash fire.

Keep away from heat, sparks, and open flame.

Keep container closed.

Use with adequate ventilation.

Avoid build up of vapours and eliminate all sources of ignition especially nonexplosion proof electrical apparatus and heaters.

Avoid prolonged breathing of vapour or spray mist.

Avoid prolonged or repeated skin contact.

In case of spillage soak up with sand or diatomaceous earth.

In case of fire use water spray, foam, dry chemical or CO_2.